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Dharmishtha H.Patel, Paresh S.Patel and Keshav C.Patel



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RESEARCH ARTICLE

SYNTHESIS OF BISAZO REACTIVE DYES AND THEIR DYEING PERFORMANCE ON VARIOUS FIBRES

Dharmishtha H.Patel¹, Paresh S.Patel^{2*} and Keshav C.Patel³

^{1,2}Narmada College of Science and Commerce, Zadeshwar, Bharuch-392011, Gujarat, India ³Department of Chemistry, Veer Narmad South Gujarat University, Surat-395007, India

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ABSTRACT

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Key words:

4,4'-methylene bis anthranilic acid, characterisation, dyeing, fastness properties, colorimetric data. A series of bisazo reactive dyes has been synthesized by coupling of tetrazotized 4,4'-methylene bis anthranilic acid with various 2-((4-amino-3-methoxy phenyl) sulphonyl)ethyl hydrogen sulphato cyanurated coupling components such as H-acid, Gamma acid, J-acid, N-Methyl J-acid, N-Phenyl J-acid, Chicago acid, S-acid, K-acid, Bronner acid, Peri acid, Laurent acid, Koch acid, Naphthionic acid and Tobias acid. They were characterized by nitrogen elemental analysis, IR and ¹HNMR spectra. The dyeing performance of all these dyes on wool, silk and cotton fibres gave fair to good light fastness, good to excellent wash fastness and rubbing fastness. Spectral properties and colorimetric data (L^{*}, a^{*}, b^{*}, c^{*}, H^{*} and K/S) have also been investigated.

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INTRODUCTION

Reactive dyes, the newest addition of existing dyes are the centre of attraction in dyestuff research (Shah K.M., 1994). These dyes are the only textile coloration products designed to furnish covalent bonds between dye and substrate during dyeing. Most of Procion dyes are water soluble, easily applied and because the reactive group may be attached to almost any coloured molecular system, can be used to produce both very bright and very dull shades of all colours. They are used for the dyeing and printing of cellulose and to a lesser extent polyamide fibres. In addition, investigations into the development of reactive dyes for polyester and polypropylene fibres have been demonstrated to the level of technical possibility but such dyes are not yet of commercial interest (Christie R. M., 2001; Rattee I.D., 1984).

Several new reactive systems have been introduced from time to time, which covers the subjects of innumerable patents and publications (JP, 1981; Seigel E., 1972; BP, 1967; EGP, 1968). Over the last decade a number of major changes have been taken place, primary aimed at minimizing the major weakness of reactive dyes for facilitating new application condition. In this period, monoazo reactive dyes (Patel *et al*, 2014; Patel *et al*, 2015; Patel *et al*, 2015) and bisazo reactive dyes (Patel *et al*, 1998; Patel *et al*, 2009; Patel *et al*, 2011)

have been established as a major group for fixation to cellulose.

MATERIALS AND METHODS

The melting points of the compounds were determined using open capillary and are uncorrected. The purity of dyes was determined by thin- layer chromatography using silica gel-G coated Al plates. IR spectra were recorded on SHIMADZU, Model: FTIR 8400S using KBr pellets. ¹H NMR spectra were obtained on Bruker Avance-II 400 NMR spectrometer using D₂O solvent and TMS as internal reference (chemical shifts in δ , ppm). Colorimetric data (L^{*}, a^{*}, b^{*}, c^{*}, H^{*} and K/S) were recorded on Reflactance spectrophotometer Gregtag Macbeth Color-Eye 7000A.

Synthesis of 4, 4'-methylene bis anthranilic acid (I)(Patel et al, 1996; Patel et al, 1999)

Anthranilic acid (13.7 g, 0.1mol) was dissolved in water (125 ml) and 36.5% hydrochloric acid (25 ml) at 50 °C and then treated with 3% aqueous formaldehyde solution (35ml). The temperature was maintained 60 °C and stirred for an hour and neutralized with 10% sodium hydroxide to give yellow precipitates of 4, 4'-methylene-bis anthranilic acid. It was filtered, washed with hot water, dried and recrystallized from ethanol. Yield: 84 %, m.p. 200-205 °C.

^{*}Corresponding author: Paresh S.Patel

Narmada College of Science and Commerce, Zadeshwar, Bharuch-392011, Gujarat, India

Tetrazotization of 4,4'-methylene bis anthranilic acid(II)

4, 4'-Methylene-bis anthranilic acid (2.86 g, 0.01 mol) was suspended in H₂O (30 ml). Conc. hydrochloric acid (10 ml) was added dropwise to this well stirred suspension. The mixture was gradually heated up to 70 °C, till clear solution was obtained. The solution was cooled to 0-5 °C in an ice bath. To this solution, NaNO₂ (1.38 g, 0.02mol) in water (5 ml) was added to the above solution at 0 °C temperature over a period of five minutes with continuous stirring. The stirring was continued for an hour, maintaining the same temperature, with positive test for nitrous acid on starch iodide paper. The excess of nitrous acid was destroyed with required amount of sulphamic acid solution in water. The clear tetrazotized solution at 0-5 °C was obtained and used for subsequent coupling reaction.

Synthesis of 2-(4-amino-3-methoxy phenyl sulphonyl)ethyl hydrogen sulphato cyanurated H-acid(III)

Cyanuration of H-acid

Cyanuric chloride (3.69 g, 0.02mol) was stirred in acetone (50 ml) at a temperature below 5 °C for a period of an hour. A neutral solution of H-acid (6.38 g, 0.02mol) in aqueous sodium carbonate solution (10% w/v) was then added in small lots in about an hour. The pH was maintained neutral by simultaneous addition of sodium carbonate solution (1% w/v). The solution was used for subsequent coupling reaction.

Condensation with 2-(4-amino-3-methoxy phenyl sulphonyl) ethyl hydrogn sulphate

The temperature of ice-cooled well stirred solution of cyanurated H-acid was gradually raised to 45 °C. 2-(4-amino-

3-methoxy phenyl sulphonyl)ethyl hydrogen sulphate (6.22 g, 0.02 mol) was added slowly to the cyanurated H-acid solution at same temperature during a period of 30 minutes, maintaining the pH neutral by simultaneous addition of sodium carbonate solution (1% w/v). After the addition was completed, stirring was continued for further 3 hours. The 2-(4-amino-3-methoxy phenyl sulphonyl) ethyl hydrogen sulphato cyanurated H-acid solution thus obtained was subsequently used for further coupling reaction.

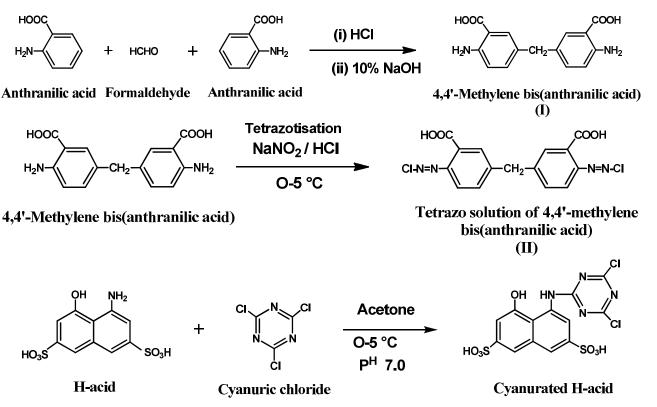
Coupling of tetrazotized solution with 2-((4-amino-3-methoxy phenyl) sulphonyl) ethyl hydrogen sulphato cyanurated H-acid coupling component

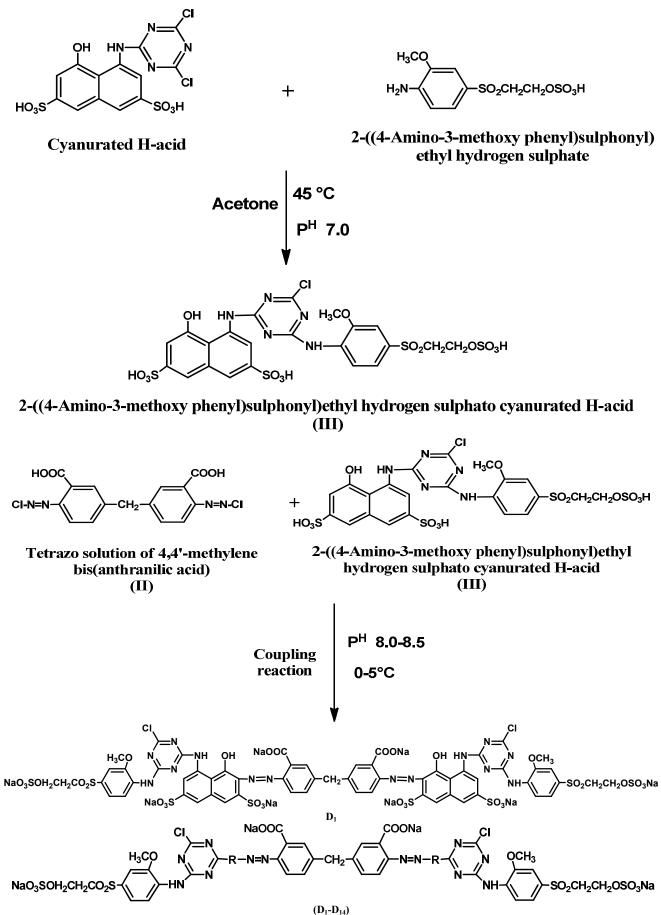
Synthesis of dyes $(D_1 to D_{14})$

Reaction Scheme

The above mentioned freshly prepared tetrazotized solution was added to an ice cooled and well stirred solution of 2-((4-amino-3-methoxy phenyl)sulphonyl) ethyl hydrogen sulphato cyanurated H-acid over a period of 10-15 minutes. The pH was maintained at 8.0 to 8.5 by simultaneous addition of sodium carbonate solution (10% w/v) and stirring was continued for 4 hours. Sodium chloride (15 g) was then added and the mixture was stirred for an hour. The solid dye separated out was filtered, washed with minimum amount of acetone and dried at room temperature.

Following the above procedure, other reactive dyes D_2 to D_{14} were synthesized by using o-anisidino cyanurated coupling components such as Gamma acid, J-acid, N-Methyl J-acid, N-Phenyl J-acid, Chicago acid, S-acid, K-acid, Bronner acid, Peri acid, Laurent acid, Koch acid, Naphthionic acid and Tobias acid.





Where, R= various coupling components to synthesise D_1 to D_{14} (Table-1)

RESULT AND DISCUSSION

Characterisation

All the synthesised dyes have been characterised by Visible absorption spectra, some representative IR spectra and PMR spectra.

Visible Absorption Spectra

The visible absorption spectra of all synthesised dyes have been recorded on in water and observed in the range of 550-383 nm. The colour observed for each dye is due to the oscillation of electrons and presence of substituents.

IR Spectra

IR spectra of dye D_1 showed O-H stretching vibration of -OH group at 3440 cm⁻¹, N-H stretching vibration of secondary amine at 3424 cm⁻¹, C-H stretching vibration of -OCH₃ group at 2960 cm⁻¹, C-H stretching vibration of -CH₂- group at 2845 cm⁻¹, C=O stretching vibration of –COOH group at 1707 cm⁻¹, C=C stretching vibration of aromatic ring at 1608 cm⁻¹, N=N stretching vibration of azo group at 1450 cm⁻¹, C-N stretching vibration of SO₂ group at 1336 and 1140cm⁻¹, S=O stretching vibration of -SO₃Na group at 1260,1160 and 1048 cm⁻¹, C-Cl stretching vibration of chloro group at 725 cm⁻¹.

IR spectra of dye D_3 showed O-H stretching vibration of -OH group at 3445 cm⁻¹, N-H stretching vibration of secondary amine at 3390 cm⁻¹, C-H stretching vibration of -OCH₃ group at 2960 cm⁻¹, C-H stretching vibration of -CH₂- group at 2842 cm⁻¹, C=O stretching vibration of –COOH group at 1707 cm⁻¹, C=C stretching vibration of aromatic ring at 1607 cm⁻¹, N=N stretching vibration of azo group at 1449 cm⁻¹, C-N stretching vibration of SO₂ group at 1330 and 1137cm⁻¹, S=O stretching vibration of -SO₃Na group at 1260,1186 and 1052 cm⁻¹, C-Cl stretching vibration of chloro group at 725 cm⁻¹.

IR spectra of dye D_4 showed O-H stretching vibration of -OH group at 3438 cm⁻¹, N-H stretching vibration of secondary amine at 3418 cm⁻¹, C-H stretching vibration of -OCH₃ group at 2940 cm⁻¹, C-H stretching vibration of -CH₂- group at 2843 cm⁻¹, C=O stretching vibration of -COOH group at 1708 cm⁻¹,

C=C stretching vibration of aromatic ring at 1607 cm⁻¹, N=N stretching vibration of azo group at 1449 cm⁻¹, C-N stretching vibration of tertiary amine group at 1533 cm⁻¹, S=O stretching vibration of SO₂ group at 1330 and 1136cm⁻¹, S=O stretching vibration of -SO₃Na group at 1257, 1160 and 1051 cm⁻¹, C-Cl stretching vibration of chloro group at 710 cm⁻¹.

¹HNMR Spectra

¹HNMR Spectra of dye D_2 showed signals at 1.94 (s, 6H, -OCH₃), 3.68-3.72 (t,4H,-CH₂-protons), 3.91 (s,2H,-CH₂-protons), 3.94 (s,2H,-NH-protons), 4.10-4.14 (t, 4H,-CH₂-protons), 4.90 (s,2H,-OH proton), and 6.78-7.80 (m,22H, aromatic protons).

Application

Dyeing of fibres

All the dyes were applied on silk, wool and cotton fibres by using the standard procedure (Shenai V.A., 1973).

Exhaustion and fixation study

The percentage exhaustion of 2% dyeing on silk fabric ranges from 68-75%, for wool ranges from 68-75% and for cotton ranges from 68-75%. The percentage fixation of 2% dyeing on silk fabric ranges from 85-90%, for wool ranges from 86-92% and for cotton ranges from 87-92% (Table-2).

Fastness properties

Fastness to light was assessed in accordance with BS: 1006-1978(Standard Test Method, 1994). The rubbing fastness test was carried out by using Crock meter (Atlas) in accordance with the AATCC-1961 (AATCC Test, 1961) and the wash fastness was carried out in accordance with IS:765-1979 (Indian Standard, 1979). All the dyes show generally fair to good light fastness properties. The washing and rubbing fastness properties ranges from very good to excellent fastness on silk, wool and cotton (Table-3).

Computer Colour Matching Data (CCM)

The colour on silk, wool and cotton fibres are expressed in terms of CIELab values and the coordinates measured are

Dye No.	Coupling	Molecular Formula	Mol. Weight	Yield	M.P.	Nitro	Rf	
•	component (R)	wolecular Formula	g /mol	(%)	°C	Found	Req.	Value
D_1	H-acid	C ₅₉ H ₄₀ N ₁₄ S ₈ O ₃₂ Cl ₂ Na ₈	1968	81	>300	9.88	9.96	0.56
D_2	Gamma acid	$C_{59}H_{42}N_{14}S_6O_{26}Cl_2Na_6$	1764	78	>300	11.02	11.11	0.60
D_3	J-acid	C59H42N14S6O26Cl2Na6	1764	84	>300	11.04	11.11	0.58
D_4	N-methyl J-acid	$C_{61}H_{46}N_{14}S_6O_{26}Cl_2Na_6$	1732	76	>300	11.02	10.94	0.62
D_5	N-phenyl J-acid	C71H50N14S6O26Cl2Na6	1916	86	>300	10.32	10.23	0.54
D_6	Chicago acid	C59H40N14S8O32Cl2Na8	1968	78	>300	9.86	9.96	0.52
D_7	S-acid	C ₅₉ H ₄₂ N ₁₄ S ₆ O ₂₆ Cl ₂ Na ₆	1764	82	>300	11.24	11.11	0.60
D_8	K-acid	C59H40N14S8O32Cl2Na8	1968	77	>300	9.85	9.96	0.57
D_9	Bronner acid	C ₅₉ H ₄₂ N ₁₄ S ₆ O ₂₄ Cl ₂ Na ₆	1732	81	>300	11.25	11.32	0.62
D_{10}	Peri acid	C59H42N14S6O24Cl2Na6	1732	79	>300	11.20	11.32	0.58
D_{11}	Laurent acid	C ₅₉ H ₄₂ N ₁₄ S ₆ O ₂₄ Cl ₂ Na ₆	1732	86	>300	11.38	11.32	0.56
D ₁₂	Koch acid	C59H38N14S10O36Cl2Na10	2140	79	>300	9.26	9.16	0.62
D ₁₃	Naphthionic acid	$C_{59}H_{42}N_{14}S_6O_{24}Cl_2Na_6$	1732	82	>300	11.23	11.32	0.54
D ₁₄	Tobias acid	$C_{59}H_{44}N_{14}S_4O_{18}Cl_2Na_4$	1528	80	>300	12.72	12.83	0.52

Table 1 Characterization of bisazo reactive dyes

lightness (L*), chroma (C*), hue angle form 0° to 360° (H), a* value represent the degree of redness (positive) and greenness (negative) and b* represents the degree of yellowness (positive) and blueness (negative).

For silk fibre, the data summarized in Table-4 showed that dye D_1 is more darker and D_{13} is more lighter. D_4 is more redder and D_{13} is more greener, D_{12} is more yellower and D_1 is more bluer, D_1 is more duller and D_4 is more brighter. D_1 has more

Dye No.	Shade on silk fabric	Shade on wool fabric	Shade on cotton fabric	λmax	%	5 Exhausti	on	% Fixation			
					S	W	С	S	W	С	
D_1	Dark maroon	Magenta	Dark violet	550	74.75	74.5	74.7	90.30	90.60	91.03	
D_2	Maroon	Dark maroon	Dark maroon	534	74.05	73.95	74.1	89.80	90.60	91.76	
D_3	Red	Oranges red	Brownish maroon	501	73.85	73.65	73.95	88.01	91.64	90.60	
D_4	Red	Red	Reddish maroon	512	73.8	69.44	73.85	87.40	90.35	90.72	
D_5	Reddish brown	Rustic brown	Light brown	522	73.7	73.5	73.80	87.40	89.80	89.43	
D_6	Dark brown	Light brown	Brownish khakhi	379	70.95	70.85	71.05	89.49	88.92	88.67	
D_7	Yellowish brown	Orangey brown	Turmeric yellow	383	71.80	71.65	71.85	89.83	88.62	89.07	
D_8	Golden yellow	Yellowish brown	Yellow	361	71.45	71.2	68.35	87.47	87.78	87.05	
D_9	Dark yellow	Lemon yellow	Pale yellow	387	68.60	68.95	69.15	88.92	88.68	88.21	
D_{10}	Pinkish brown	Brick red	Light brown	385	70.4	70.25	70.45	85.23	86.83	88.71	
D ₁₁	Pale yellow	Pale yellow	Yellow	388	68.7	68.50	71.0	88.79	87.59	89.44	
D ₁₂	Golden yellow	Dark yellow	Dark yellow	386	69.20	68.90	70.15	88.15	87.80	88.38	
D ₁₃	Light yellow	Light yellow	Pale yellow	391	68.45	68.35	69.85	89.85	87.88	87.32	
D ₁₄	Light brown	Pale yellow	Yellow	386	69.25	68.95	69.60	90.25	88.46	88.36	

Table 2 Dy	eing perfo	ormance of	Dyes	D_1	to D ₁₄
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S= silk, W= wool and C=cotton

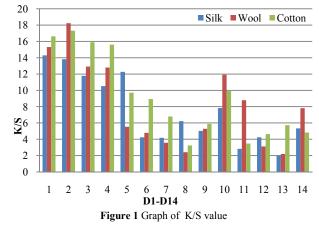
Table 3 Fastness properties of Dyes D_1 to D_{14}

D	Lial	t for	tra o a a	Wa	sh fas	troop		R	lubbii	ng fast	g fastness				
Dye No	Ligi	it fas	tness	vv a	sn ias	uness		Dry			Wet				
110.	S	W	С	S	W	С	S	W	С	S	W	С			
D_1	5-6	5-6	5	4-5	4-5	4	4	4	4-5	4	4-5	4-5			
D_2	5-6	5	5	4	4	3-4	5	5	4	4	3-4	4-5			
D_3	4-5	5	4-5	4-5	5	4	4	4	4	4-5	3-4	4			
D_4	4-5	5	4	4	4	4	4-5	4	3-4	3-4	4	3-4			
D_5	3-4	5	4	3-4	3	3	3	4-5	3-4	3	4	3-4			
D_6	4-5	4-5	4-5	4	4	3	4	4	4	3-4	4-5	3			
D_7	4-5	4-5	3-4	4-5	4	3-4	5	3-4	4	4	3	3			
D_8	4-5	5	4	4-5	4	4	4	4-5	3-4	3-4	4-5	3-4			
D_9	5-6	5	5	4-5	4	4-5	4	4	3-4	4	4	4			
D_{10}	4-5	4-5	5	4	4-5	4	4	5	4	4	4-5	4			
D ₁₁	4	5	4-5	4	4-5	3-4	4	3-4	4	4	4	3-4			
D ₁₂	5	4-5	5	5	4	3-4	4	4	3-4	3-4	3-4	4			
D ₁₃	4-5	5	4-5	3-4	4	4	4	3-4	4	4	4-5	3-4			
D ₁₄	4-5	5	5	4	4-5	4	4	4-5	4	4-5	4	4			

S= silk, W= wool and C=cotton; Light fastness: 1-Poor, 2-Slight,3-Moderate, 4-Fair , 5-Good, 6-Very good,7-Excellent,8-Maximu; Wash and Rubbing fastness: 1-Poor, 2-Fair,3-Good, 4-Very good, 5-Excellent

K/S values given by the reflectance spectrophotometer were calculated at λ max and are directly correlated with the dye concentration on the substrate according to the Kubelka–Munk equation (Billmeyer *et al*, 1981; Volz H.G., 1995). The colorimetric data of silk, wool and cotton fibres for all dyes are summarised in Table-4. The K/S value graph of all the dyes is shown in Figure-1 and graph of b* versus a* for silk, wool and cotton fibres are shown in Figure-2, 3 and 4 respectively.

K/S value and D₁₃ has less K/S value.



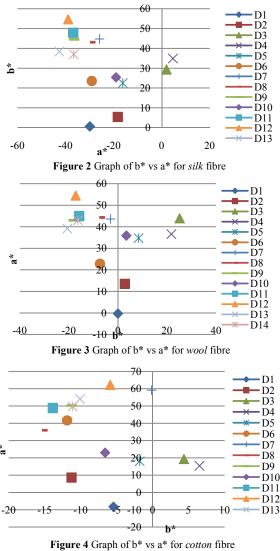
For wool fibre, the data showed that dye D_1 is more darker and D_{13} is more lighter. D_3 is more redder and D_{13} is more greener, D_{12} is more yellower and D_1 is less yellower, D_6 is more duller and D_4 is more brighter. D_2 has more K/S value and D_{13} has less K/S value.

For cotton fibre, the data showed that dye D_2 is more darker and D_8 is more lighter. D_2 is more redder and D_4 is more greener, D_{12} is more yellower and D_1 is more bluer, D_2 is more duller and D_{12} is more brighter. D_2 has more K/S value and D_8

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Dye		L*		a*			b* C*					H*				K/S		
No.	S	W	С	S	W	С	S	W	С	S	W	С	S	W	С	S	W	С
D_1	-18.04	0.15	-0.16	-29.91	-0.02	-5.44	0.65	-0.3	-8.22	-29.59	0.1	0.45	-4.39	-0.28	-9.85	14.28	15.31	16.63
D_2	-0.55	-2.25	-9.85	-18.29	2.78	-11.27	5.45	13.5	8.64	-18.45	1.69	-12.94	4.91	13.68	5.84	13.81	18.23	17.32
D_3	5.99	12.55	-5.13	2	25.13	4.35	29.38	43.98	19.36	8.77	35.92	5.09	28.11	35.72	19.18	11.78	12.91	15.89
D_4	4.92	10.84	-3.29	4.66	21.77	6.51	34.92	36.61	15.43	13.99	28.84	5.79	32.33	31.35	15.71	10.52	12.8	15.61
D_5	16.04	17.95	1.46	-16.21	8.4	-1.8	22.52	34.76	18.05	-10.21	17.22	-1	25.8	31.34	18.11	12.27	5.53	9.72
D_6	12.25	20.8	33.05	-29.1	-7.38	-11.88	23.58	22.86	41.71	-18.15	-2.3	12.95	32.76	23.91	41.39	4.23	4.79	8.93
D_7	25.48	35	31.16	-25.86	-3.05	-0.15	44.81	43.59	59.2	2.51	17.65	33.16	51.68	39.97	49.05	4.19	3.57	6.79
D_8	30.47	36.95	52.26	-29.67	-7.44	-15.31	43.1	44.32	35.99	-0.28	16.68	6.68	52.32	41.72	38.54	6.23	2.41	3.23
D_9	34.58	51.56	45.31	-36.4	-18.07	-11.25	44.6	43.04	50.34	-0.08	13.41	21.52	57.57	44.71	46.88	5.02	5.27	5.89
D_{10}	20.25	33.48	31.87	-19.01	3.4	-6.6	25.42	35.89	23.03	-10.42	14.61	-1.66	29.98	32.95	23.9	7.83	11.92	9.92
D_{11}	39.76	46.02	49.18	-36.82	-15.82	-13.87	47.92	44.98	48.88	3.18	15.53	19.63	60.34	45.09	46.86	2.82	8.79	3.47
D_{12}	36.79	45.22	41.37	-38.92	-17.26	-5.89	54.69	54.52	62.31	9.82	24.92	34.38	66.4	51.47	52.3	4.23	3.12	4.64
D ₁₃	43.47	52.97	47.44	-42.57	-20.64	-10.09	38.41	39.11	54.07	-6.44	9.46	25.4	56.97	43.2	48.79	2.03	2.19	5.73
D_{14}	39.58	48.33	49.66	-36.61	-16.33	-11.12	37.02	43.03	49.8	-7.63	13.53	21.01	51.5	43.99	46.5	5.34	7.81	4.82

Table 4 Color measurement (CIELab) data of Dyes D_1 to D_{14}

has less K/S value.



CONCLUSION

4,4'-Methylene bis anthranilic acid was tetrazotised and coupling with various 2-((4-amino-3-methoxy phenyl) sulphonyl) ethyl hydrogen sulphato cyanurated coupling components gave corresponding bisazo reactive dyes (D_1 to D_{14}). These dyes gave violet to yellow shade on silk, wool and cotton fibres and showed fair to good light fastness, good to excellent fastness to washing and rubbing fastness. Exhaustion and fixation of these dyes are very good in order. The presence of triazine group of dye molecule improves the exhaustion, fixation and fastness properties. D_1 has highest K/S value for silk fibre and D_2 has highest K/S value for wool and cotton fibre. Thus, D_1 has more dye concentration on silk fabric and D_2 has more dye concentration on wool and silk fibre.

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